HPLC reverse phase system using a nonpolar 25 cm x 4.6 mm ID LC-18 column with acetone-acetonitrile (1:1, v/v) as the mobile phase at a flow rate of 1 ml/min and refractive index detection. The methods developed were then used to separate the dimeric fraction of differently oxidized soybean oil samples into their component dimers which were isolated and structually characterized with the aid of GC-MS. The presence of several dimers containing hydroxy, keto, unsaturated monocyclic, bicyclic and noncyclic as well as saturated tricyclic structuures was indicated and related to oxidation parameters.

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Quantitative Determination of Monomeric Cyclic Fatty Acids in Fats and Oils Analytical Consideration. J.A. Rojo, University of Illinois, 1208 W. Pennsylvania Ave., Urbana, IL 61801, and E.G. Perkins, University of Illinois.

The current analytical methodologies for the determination of monomeric cyclic fatty acids (EFA) in edible fats and oils follow the same basic principle: separation and concentration of CFA containing fractions from methylated-hydrogenated oils, and quantitation by gas chromatography (GC). Their major limitations are high variability resulting from several successive operations involved, the complex mixture of substances present in the CFA fractions, lengthy procedures, and the total uncertainty associated with the identity and structure of the analites. A faster and more reliable procedure has been developed by simplifying the number of operations and by statistical optimization of the analytical conditions. Clean up of the CFA fraction by solid phase extraction (SPE) previous high resolution GC is described. Confirmation of CFA peaks is carried out using GC-MS. The introduction of an empirical methematical model allowed the prediction of the GC elution pattern of isomeric CFA's and facilitated their individual structure identification and quantitation.

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High Performance Liquid Chromatographic Analyses of Mixed Glycerol-Ether-Esters. Thomas A. Foglia, Eastern Regional Research Center, ARS/USDA, 600 E. Mermaid Lane, Philadelphia, PA 19118, and Peter Vail and Philip Sonnet.

Mixed alkyl-acyl-glycerols can serve as useful substrates in defining the positional selectivity of lipases. Additionally, such compounds also have found utility in studies on transport properties of lipids, i.e., intestinal fat absorbtion. The synthesis of alkyl-acyl-glycerols has been described, but such procedures often lead to isomeric mixtures which are difficult to separate. Moreover, for biological studies mixed ether ester glycerols having equivalent chain lengths are more desirable. However, the composition of such mixtures is the more difficult to determine. With recent advances in high performance liquid chromatography (HPLC), many of the separation problems previously encountered have been overcome. In this paper we will present data on both the normal and reverse phase HPLC separation of a series of isomeric mixed alkyl-acyl-glycerols of equivalent and non-equivalent chain lengths.

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Separation and Determination of Hydroperoxides From Oxidized Triglycerides Using HPLC. Elizabeth M. Kay, University of Saskatchewan, Department of Crop Science and Plant Ecology, Saskatoon, Sasketchewan, S7N 0W0 Canada, and Frank W. Sosulski and Alan R. McCurdy, University of Sasketchewan.

A high performance liquid chromatography (HPLC) method has been developed to separate and determine hydroperoxides of oxidized triglycerides and to determine the degree of oxidation of oil samples. The oxidized samples were reduced to hydroxy derivatives using sodium borohydride. These derivates were injected onto a normal phase column (Partisil 5) and separated using 1% isopropyl alcohol in hexane as eluant with UV detection at 234 nm. Individual triglycerides were separated into a number of peaks on the basis of hydroperoxy position and conjugated double bond configuration, but not by double bond number, with the result that some overlap existed between linolein and linolenin isomers. It was possible to separate oxidized derivatized canola oil into 10-20 peaks corresponding to peaks from individual triglycerides; this separation made it possible to observe changes in isomer distribution during oxidation. Good correlations (greater than 0.995) were found between total oxidized area from the HPLC chromatogram and peroxide value of the samples. From this preliminary work it appears that this simple HPLC technique can be used to follow the course of oxidation in oil samples, and to yield valuable information about changes in hydroperoxides during oxidation.

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Discrimination Between Oxidation and Maillard Browning by Means of Fluorescence in Model Systems and Foods. William L. Porter, Natick RD & E Center, Kansas Street, Natick, MA 01760-5020, and E.D. Black, Y-D. Kim, L. Hoke and J.G. Kapsalis, U.S. Army Natick RD & E Center.

Energy-dense combat ration model systems containing lactose, casein and stripped corn oil encapsulated in acetone-stripped soy lecithin have been monitored during storage at high temperatures for polymerization by means of absorption and fluorescence spectrophotometry. The objective was to develop rapid, labor-saving methods applicable to both soluble and heavily cross-linked, insoluble components of the heated model system. We have shown that in our system production of color, fluorescence and high molecular weight materials are closely correlated. Methods to monitor sugar-amine (Maillard) browning include 410 nm absorption and solution fluorescence of the aqueous phase after pronase digest, solution fluorescence of the chloroform-methanol extract, and front-face fluorescence of acid-precipitated casein slurries from the lipid-free browned system. Lipid oxidation monitoring methods include vapor phase detection by means of polyamide plate fluorescence in the presence of oxidizing lipid, chloroform-methanol extract fluorescence, hexane extract absorption at 234 nm, and front-face fluorescence of acid-precipitated casein slurries. Excitation and emission wavelengths of the Maillard-derived and oxidation-derived chloroform-methanol extract fluorescence are initially quite similar, but products of the two processes can be distinguished because at equal levels of extract fluorescence intensity, oxidized samples have much higher Hunter reflectance L and b values, whereas Maillard-browned samples have higher Hunter a, lower b and much lower L. An additional distinction is that polyamide plates in the vapor phase produce substantial fluorescence only over oxidizing systems. The chloroform-methanol, front-face, and polyamide fluorescence methods are relatively free from interference arising from common food components. They are potentially widely applicable, rapid, and susceptible to semi-automation.

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A Rapid Method for the Determination of Total Polar Components in Frying Oils. Sharon L. Melton, University of Tennessee, Food Technology and Science, P.O. Box 1071, Knoxville, TN 37901, and Danielle Sykes, University of Tennessee.

At the present time, the best method for measurement of total polar components (TPC) in frying oils is the IUPAC method. However, this method requires approximately 3 hr/sample; it is too long to be useful in quality control, and a rapid method needs to be developed. In developing the method, disposable silica columns from different manufacturers were tried with different eluting solvents to separate the nonpolar components (NPC) from TPC in used frying oils that contained from 4.6 to 36.6% TPC. The efficiency of separation of TPC from NPC and characterization of